

# CODING FORM FOR SRC INDEXING

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| Submitting Organization  |               |              |
| ICI AMERICAS INC   |               |              |
|  |               |              |
| Contractor   |               |              |
| IMPERIAL CHEM INDUS LAB  |               |              |
|  |               |              |
| Document Title   |               |              |
| <p>RUBINATE M: ACUTE TOXICITY OF THE PRODUCTS OF HYDROLYSIS TO<br/>         FATHEAD MINNOW (PIMEPHALES PROMELAS) WITH COVER LETTER DATED<br/>         051992</p> |               |              |
|  |               |              |
| Chemical Category  |               |              |
| RUBINATE M (9016-87-9)   |               |              |



# Polyurethanes

CONTAINS NO CBI

94 MAY 22 11 2:05

May 19, 1992

## ICI Polyurethanes Group

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Environmental Protection Agency  
401 M Street, S.W.  
Washington, DC 20460

86920000936



86920000936

ATTN: Section 8(d) Reporting

Dear Sir/Madam:

The enclosed report is submitted in accordance with the requirements of TSCA Section 8(d) as specified in 40 CFR Part 716. This report represents a study recently made available to ICI Americas, Inc., by our parent company (ICI-PLC) in the United Kingdom.

The following listed report is enclosed for your review.

Chemical

CAS Number  
9016-87-9

Report Date  
February 1992

Title  
RUBINATE M: Acute Toxicity of the  
products of hydrolysis to fathead  
minnow (Pimephales promelas)

Note: The test laboratory did not correctly describe the material tested in the summary (Page 5) and Materials (Page 6)

### INCORRECT DESCRIPTION:

Synonyms: Blend of Polymeric and 4,4' MDI  
Polymeric MDI (CAS 9016-87-9)  
4,4' MDI (CAS 101-68-8)

### CORRECT DESCRIPTION:

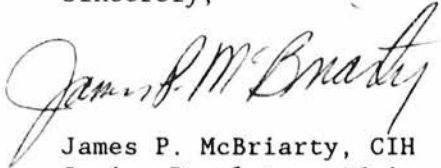
Synonyms: Polymeric MDI (CAS 9016-87-9) 100%  
contains  
4,4' -MDI (CAS 101-68-8) ~50%

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92 N.Y.C. 84-2-88  
This discrepancy has been noted on the appropriate pages of the report and  
bear my initials JMcB.

If you have any questions please contact me at (609) 423-8356.

Sincerely,



James P. McBriarty, CIH  
Senior Regulatory Advisor

JPM/tas  
05199202  
attachment

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22 MAY 23 PM 2:05

GROUP ENVIRONMENTAL  
LABORATORY

# REPORT

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BL4441/B

Copy number 11

"RUBINATE" M: Acute toxicity of the products of  
hydrolysis to fathead minnow (*Pimephales promelas*)

22 MAY 23 PM 2:05  
BRITISH AM



ICI Group Environmental Laboratory  
Freshwater Quarry Bridgman Devon TQ5 8BA  
Telephone 0603 882862

Performing laboratory project identification W013/A

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MAY 06 1992

SHEA, WEST DEPTFORD  
ICI POLYURETHANES

BL4441/B

Copy number 11

"RUBINATE" M: Acute toxicity of the products of  
hydrolysis to fathead minnow (*Pimephales promelas*)

Data requirements

California Department of Health  
Services identification of hazardous  
wastes.

Study completed

28 February 1992

Performing laboratory

Imperial Chemical Industries PLC  
Group Environmental Laboratory  
Brixham Devon TQ5 8BA  
UK

Sponsor

ICI Polyurethanes

Authors

D Brown  
J E Caunter  
B G Maddock

Approved by

L F Reynolds  
April 1992

BL4441/B

"RUBINATE" M: Acute toxicity of the products of  
hydrolysis to fathead minnow (*Pimephales promelas*)

92 MAY 20 PM 2:05

Brixham study number W013/A

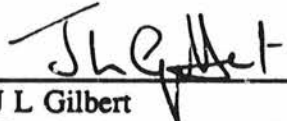
QUALITY ASSURANCE STATEMENT

The conduct of this study has been inspected/audited in accordance with Imperial Chemical Industries' policies and procedures for Good Laboratory Practice, as follows

| <u>Date</u>   | <u>Inspection/audit</u> | <u>Date of QA report</u> |
|---------------|-------------------------|--------------------------|
| 14 April 1992 | Draft report            | 14 April 1992            |
| 30 April 1992 | Final report            | 30 April 1992            |

Facilities and procedures associated with this type of study are periodically inspected in accordance with QA Standard Operating Procedures.

So far as can be established, the methods described and the results incorporated in the final report accurately reflect the raw data produced during the study.

  
J L Gilbert  
Quality Assurance Unit

30.4.92  
Date



BL4441/B

"RUBINATE" M: Acute toxicity of the products of  
hydrolysis to fathead minnow (*Pimephales promelas*)

Brixham study number W013/A

# AUTHENTICATION STATEMENT

I, the undersigned, hereby declare that this study was performed under my direction according to the principles of Good Laboratory Practice and that this report represents a true and accurate record of results obtained.

Study Director

B G Maddock  
B G Maddock

27-4-92  
Date

The following personnel carried out work on this study:

Principal Investigator

J E Caunter  
J E Caunter

27.4.92  
Date

Report approved by  
Programme Manager

L F Reynolds  
L F Reynolds

27/4/92  
Date

BL4441/B

"RUBINATE" M: Acute toxicity of the products of  
hydrolysis to fathead minnow (*Pimephales promelas*)

Brixham study number W013/A

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## 1 SUMMARY

Sponsor ICI Polyurethanes

Contacts Mrs J M Dobbs ICI OHD Polyurethanes, S.I.E. Dept, Everberg, Belgium  
Tel (32)-27589311  
R A LaBonte ICI Polyurethanes, West Detford, USA  
Tel (1)609 423-8413

Location of study raw data Imperial Chemical Industries PLC, Group Environmental Laboratory, Brixham, Devon TQ5 8BA, UK

Test material "Rubinate" M

Note: The test laboratory did not correctly describe the material tested in the summary (Page 5) and Materials (Page 6)

Synonyms Blend of Polymeric and 4,4' MDI  
Polymeric MDI (CAS No 9016-87-9)  
4,4' MDI (CAS No 101-68-8)

INCORRECT DESCRIPTION

Synonyms Blend of Polymeric and 4,4' MDI  
Polymeric MDI (CAS 9016-87-9)  
4,4' MDI (CAS 101-68-8)

CORRECT DESCRIPTION

Synonyms Polymeric MDI (CAS 9016-87-9) 100%  
contains  
4,4'-MDI (CAS 101-68-8) ~50%

Subject Acute toxicity to fathead minnow (*Pimephales promelas*)

Test concentrations Dilution water control and 250, 500 and 750 mg/l in duplicate (nominal concentration "Rubinate" M).

Duration of test 16 hours hydrolysis  
96 hours fish exposure, static with aeration.

Test date 24-28 February 1992

Test temperature 20.0 ± 1.0°C

Results No effects observed at maximum concentration of 750 mg/l (nominal concentration "Rubinate" M).

Test species Fathead minnow (*Pimephales promelas*) batch reference 1099B

Source of test animals SP Engineering, PO Box 848, Salem, MA 01970, USA

Weight/length of control fish Control<sup>1</sup>A Weight: range 0.39 - 0.43 g, mean 0.41 g  
Length: range 30 - 33 mm, mean 30.7 mm  
Control<sup>1</sup>B Weight: range 0.41 - 0.52 g, mean 0.46 g  
Length: range 30 - 33 mm, mean 31.6 mm

## 2 INTRODUCTION

At the request of ICI Polyurethanes the acute toxicity of the hydrolysis products of "Rubinate" M to fathead minnow (*Pimephales promelas*) was determined in freshwater.

The definitive study was undertaken between 24 and 28 February 1992. The study number was W013/A and the Brixham sample number was W013.

All original data together with other documents relevant to this study are filed in the ICI Group Environmental Laboratory archive at Brixham.

## 3 MATERIALS

### 3.1 Test material

The test material "Rubinate" M, was supplied by ICI Polyurethanes through Rubicon Inc, River Road, Geismar, LA 70734 USA.

Known synonyms: Blend of Polymeric and 4,4' MDI  
Polymeric MDI (CAS No 9016-87-9)  
4,4' MDI (CAS No 101-68-8)

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#### CORRECT DESCRIPTION

Synonyms: Polymeric MDI (CAS 9016-87-9) 100%  
contains  
4,4'-MDI (CAS 101-68-8) ~50%

The sample was received at ICI Group Environmental Laboratory, Brixham on 9 January 1992 and was assigned the Brixham test substance reference number W013. The material was a dark brown viscous liquid.

### 3.2 Test species

Fathead minnow (*Pimephales promelas*) was used as the test species. The fish were obtained from SP Engineering, Box 848, Salem, MA 01970, USA on 22 January 1992. There were no deaths during the four weeks leading up to the start of the study and they received no medication. The pretest diet was BP Mainstream (batch ref. FF156), a proprietary product and frozen brine shrimp (batch ref. FF152). The fish were not fed during the test or for a period of two days prior to the start of the test. The batch of fish used in this study (batch ref. 1099B) was held for 10 days at  $20 \pm 1^\circ\text{C}$  before the start of the test.

At the end of the exposure period the fish from the control tanks were weighed and measured. For control A the range in weight was 0.39 to 0.43 g with a mean of 0.41 g and the range in length was 30 to 33 mm and mean of 30.7 mm. For control B the range in weight was 0.41 to 0.52 g with a mean of 0.46 g and the range in length was 30 to 33 mm with a mean of 31.6 mm.

Twenty fish were exposed at each concentration and control, ten in each duplicate test vessel. The highest loading was 0.46 g/l and the largest fish was 1.33 times that of the smallest.

## 4 TEST METHOD AND CONDITIONS

### 4.1 Bioassay test method

The bioassay test method used in this study was based on the California Department of Fish and Game Water Pollution Control Laboratory Guideline 'Static Acute Bioassay Procedures for Hazardous Waste Samples' prepared by James M Polisini and Rebecca G Miller, last revision November 1988. The procedure used was the ICI Group Environmental Laboratory standard operating procedure BA 098 version 01.

### 4.2 Apparatus

The test system used in this study was a static system. The test vessels were made of glass and of external dimensions 300 x 200 x 200 mm with a working volume of 10 litres. Continuous aeration in each test vessel was achieved by passing compressed air through a small bore tube.

Due to the potentially hazardous nature of the test material the preparation of the test solutions and the exposure to fish were carried out in an enclosed environment in which the air temperature was controlled to ensure the test solutions remained within the specified range of  $20 \pm 1^\circ\text{C}$ .

The dilution water was dechlorinated tap water. It was passed through activated carbon and treated with sodium thiosulphate to remove the residual chlorine. The pH was adjusted and the treated water held in a secondary reservoir. The chlorine free water was then passed through an ultra violet steriliser before being distributed to the laboratories.

### 4.3 Preparation of hydrolysis products and test solutions

Due to the test material reacting with water to produce carbon dioxide gas the recommended method of shaking in a sealed vessel was not applicable and the following procedure was used. Two litres of dilution water was added to each of six 3 litre glass beakers. Stirrers were placed in each beaker and their speed adjusted so the vortex reached the impeller. Using the density of the test material as 1.2 the required volume of test material for ten litres of solution was slowly added to each beaker: 2.1 ml = 250 mg/l, 4.2 ml = 500 mg/l and 6.25 ml = 750 mg/l. The test material formed small pale yellow droplets in the water with some material adhering to the bottom of the beaker at the highest concentration. The solutions were stirred continuously for sixteen hours, after which the test material appeared more granular, paler in colour and was adhering to the impellers and sides of the beakers.

Eight litres of dilution water were added to each of the test vessels and the appropriate beaker, its contents and the impeller were added to the test vessels. Each concentration and control were prepared and run in duplicate.

#### 4.4 Physical/chemical parameters

Measurements undertaken at the start of test and daily were; temperature, dissolved oxygen and pH. The water hardness, conductivity and alkalinity were determined in the control and highest test vessel at the start and end of the test. Measurements of pH were carried out with a Corning Model 240 meter. Dissolved oxygen values were measured with a Yellow Springs Instruments dissolved oxygen meter Model 51B. Temperature values were determined by a mercury-in-glass thermometer calibrated to 0.1°C. Conductivity was measured with a Jenway 4010 conductivity meter. Hardness was determined by titration with standard EDTA solution using BDH total hardness indicator, and alkalinity was measured by an electrometric method.

The photoperiod in this study was 16 hours light and 8 hours darkness with a 10 minute transition period between them.

#### 4.5 Observations of mortalities and symptoms of toxicity

Observations of mortalities and symptoms of toxicity were made at 24, 48, 72 and 96 hours exposure.

### 5 RESULTS

#### 5.1 Mortalities and symptoms of toxicity

Observations of mortalities are shown in Table 1, and observations of symptoms of toxicity are reported in Table 2.

No mortalities or symptoms of toxicity were recorded in any of the test concentrations.

#### 5.2 Physical/chemical parameters

During the test the following ranges of dissolved oxygen concentration, pH and temperature were recorded in the test vessels (including the controls):

|                  |                |
|------------------|----------------|
| Dissolved oxygen | 8.4 - 9.2 mg/l |
| pH               | 7.24 - 7.97    |
| Temperature      | 19.0 - 20.0°C  |

The full results are given in Tables 3 - 5

The total hardness and alkalinity values of the water in the control and highest concentration were:

|                | Control<br>(A replicate) | 750mg/l<br>(A replicate)              |
|----------------|--------------------------|---------------------------------------|
|                | 0hrs 96hrs               | 0hrs 96hrs                            |
| Total hardness | 46 - 42.7                | 47 - 44.3 mg/l as CaCO <sub>3</sub>   |
| Alkalinity     | 22.0 - 23.2              | 22.4 - 24.2 mg/l as CaCO <sub>3</sub> |
| Conductivity   | 219 - 229                | 223 - 231 µS/cm                       |

TABLE 1

"RUBINATE" M: ACUTE TOXICITY TO FATHEAD MINNOW  
SUMMARY OF PERCENTAGE MORTALITY

| Nominal concn<br>of "Rubinate" M<br>n.g/l | Percentage mortality observed |          |          |          |
|---|-------------------------------|----------|----------|----------|
|   | 24 hours                      | 48 hours | 72 hours | 96 hours |
| 250 (A)                                   | 0                             | 0        | 0        | 0        |
| 250 (B)                                   | 0                             | 0        | 0        | 0        |
| 500 (A)                                   | 0                             | 0        | 0        | 0        |
| 500 (B)                                   | 0                             | 0        | 0        | 0        |
| 750 (A)                                   | 0                             | 0        | 0        | 0        |
| 750 (B)                                   | 0                             | 0        | 0        | 0        |
| Control A                                 | 0                             | 0        | 0        | 0        |
| Control B                                 | 0                             | 0        | 0        | 0        |

TABLE 2

**"RUBINATE" M: ACUTE TOXICITY TO FATHEAD MINNOW****SUMMARY OF SYMPTOMS OF TOXICITY**

| Nominal concn<br>of "Rubinate" M<br>mg/l | Exposure time in hours |    |    |    |
|--|------------------------|----|----|----|
|  | 24                     | 48 | 72 | 96 |
| 250 (A)                                  | A                      | A  | A  | A  |
| 250 (B)                                  | A                      | A  | A  | A  |
| 500 (A)                                  | A                      | A  | A  | A  |
| 500 (B)                                  | A                      | A  | A  | A  |
| 750 (A)                                  | A                      | A  | A  | A  |
| 750 (B)                                  | A                      | A  | A  | A  |

A = No significant effect. 10% or less of test population dead or exhibiting symptoms of toxicity.



TABLE 3

"RUBINATE" M: ACUTE TOXICITY TO FATHEAD MINNOW  
SUMMARY OF DISSOLVED OXYGEN IN EXPOSURE VESSELS

| Nominal<br>concn of<br>"Rubinate" M<br>mg/l | Dissolved oxygen mg/l |       |       |       |       |
|---|-----------------------|-------|-------|-------|-------|
|   | day 0                 | day 1 | day 2 | day 3 | day 4 |
| 250 (A)                                     | 9.0                   | 9.0   | 9.0   | 9.2   | 9.2   |
| 250 (B)                                     | 9.0                   | 9.0   | 8.8   | 9.0   | 8.8   |
| 500 (A)                                     | 9.0                   | 9.2   | 9.0   | 9.0   | 9.2   |
| 500 (B)                                     | 9.0                   | 8.8   | 9.0   | 9.0   | 9.0   |
| 750 (A)                                     | 9.2                   | 9.2   | 9.2   | 9.0   | 9.0   |
| 750 (B)                                     | 9.0                   | 8.8   | 8.6   | 8.4   | 9.0   |
| Control (A)                                 | 9.2                   | 9.0   | 9.0   | 9.0   | 9.2   |
| Control (B)                                 | 9.2                   | 9.0   | 9.0   | 9.0   | 9.2   |

TABLE 4

"RUBINATE" M: ACUTE TOXICITY TO FATHEAD MINNOW  
SUMMARY OF pH IN EXPOSURE VESSELS

| Nominal<br>concn of<br>"Rubinate" M<br>mg/l | pH values |       |       |       |       |
|---|-----------|-------|-------|-------|-------|
|   | Day 0     | Day 1 | Day 2 | Day 3 | Day 4 |
| 250 (A)                                     | 7.66      | 7.85  | 7.91  | 7.42  | 7.89  |
| 250 (B)                                     | 7.65      | 7.81  | 7.85  | 7.40  | 7.85  |
| 500 (A)                                     | 7.65      | 7.76  | 7.81  | 7.38  | 7.83  |
| 500 (B)                                     | 7.63      | 7.70  | 7.72  | 7.34  | 7.77  |
| 750 (A)                                     | 7.65      | 7.76  | 7.79  | 7.39  | 7.83  |
| 750 (B)                                     | 7.72      | 7.71  | 7.72  | 7.24  | 7.79  |
| control (A)                                 | 7.60      | 7.92  | 7.97  | 7.42  | 7.94  |
| control (B)                                 | 7.64      | 7.92  | 7.94  | 7.45  | 7.93  |

TABLE 5

"RUBINATE" M: ACUTE TOXICITY TO FATHEAD MINNOW  
SUMMARY OF TEMPERATURE IN EXPOSURE VESSELS

| Nominal<br>concn of<br>"Rubinate" M<br>mg/l | Temperature °C |       |       |       |       |
|---|----------------|-------|-------|-------|-------|
|   | Day 0          | Day 1 | Day 2 | Day 3 | Day 4 |
| 250 (A)                                     | 19.2           | 19.6  | 19.8  | 19.7  | 19.6  |
| 250 (B)                                     | 19.0           | 19.6  | 19.8  | 19.8  | 19.7  |
| 500 (A)                                     | 19.2           | 19.3  | 19.5  | 19.5  | 19.3  |
| 500 (B)                                     | 19.2           | 19.8  | 19.9  | 19.9  | 19.7  |
| 750 (A)                                     | 19.2           | 19.7  | 19.8  | 19.8  | 19.6  |
| 750 (B)                                     | 19.3           | 19.8  | 20.0  | 20.0  | 19.8  |
| control (A)                                 | 19.4           | 19.7  | 19.7  | 19.7  | 19.5  |
| control (B)                                 | 19.4           | 19.5  | 19.6  | 19.6  | 19.5  |

BL4441/B

"RUBINATE" M: Acute toxicity of the hydrolysis products to fathead minnow (*Pimephales promelas*)

## CIRCULATION

Copy  
number

|       |                |   |
|-------|----------------|---|
| 1-3   | Dr J P Lyon    | ICI Americas S.H.E. Dept, Wilmington, USA         |
| 4-6   | Mrs J M Dobbs  | ICI Polyurethanes, S.H.E. Dept, Everberg, Belgium |
| 7     | Dr D Brown     | ICI Group Environmental Laboratory, Brixham       |
| 8     | Mr B G Maddock | " " " " "   |
| 9     | Mr J E Caunter | " " " " "   |
| 10-12 | Report Centre  | " " " " "   |

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